AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Preparation of Pressed Powder White Reflectance Factor Transfer Standards for Hemispherical and Bi-Directional Geometries[1]

This standard is issued under the fixed designation E 259; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

The internationally accepted standard of reflectance is the perfect reflecting diffuser. This ideal reflecting surface reflects 100 % of the incident radiant power such that the radiance is the same for all directions within the hemisphere above the surface. No physical realization of this standard exists. Optical properties of standards prepared from pressed plaques of magnesium carbonate ($MgCO_3$), barium sulfate ($BaSO_4$), or polytetrafluoroethylene (PTFE) can approximate an ideal standard. For further information see CIE Publication No. 46 **(1)**.[2] The principal use of a white reflectance factor standard is to transfer an absolute scale of reflectance to a more durable material or from one instrument to another. In theory, it should be easy to do this transfer from first principles. In practice, one is likely to need values for parameters that are unknown, proprietary, or require a high level of skill. Some, but not all, of those parameters are discussed in this practice.

1. Scope

1.1 This practice covers procedures for preparing pressed powder transfer standards. These standards can be used in the near-ultraviolet, visible and near-infrared region of the electromagnetic spectrum. Procedures for calibrating the reflectance factor of materials on an absolute basis are contained in CIE Publication No. 44 **(2)**. Pressed powder standards are used as transfer standards for such calibrations because they have a high reflectance factor that is nearly constant with wavelength, and because the geometric distribution of reflected flux resembles that from the perfect reflecting diffuser.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 284 Terminology of Appearance[3]

3. Terminology

3.1 Terms and definitions in Terminology E 284 are applicable to this practice.

3.2 *Definitions*—The following definitions are particularly important to this practice:

3.2.1 *perfect reflecting diffuser*—ideal reflecting surface that neither absorbs nor transmits light, but reflects diffusely, with the radiance of the reflecting surface being the same for all reflecting angles, regardless of the angular distribution of the incident light. (1990)

3.2.2 *reflectance*, ρ, *n*—ratio of the reflected radiant or luminous flux to the incident flux in the given conditions. [CIE][A]

3.2.3 The term *reflectance* is often used in a general sense or as an abbreviation for *reflectance factor*. Such usage may be assumed unless the above definition is specifically required by the context. (1989b)

3.2.4 *reflectance factor, R, n*—ratio of the flux reflected from the specimen to the flux reflected from the perfect reflecting diffuser under the same geometric and spectral conditions of measurement. [CIE][B](1988)

4. Summary of Practice

4.1 Procedures are given for the preparation of white

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[1] This practice is under the jurisdiction of ASTM Committee E-12 on Color and Appearance and is the direct responsibility of Subcommittee E12.02 on Spectrophotometry and Colorimetry.

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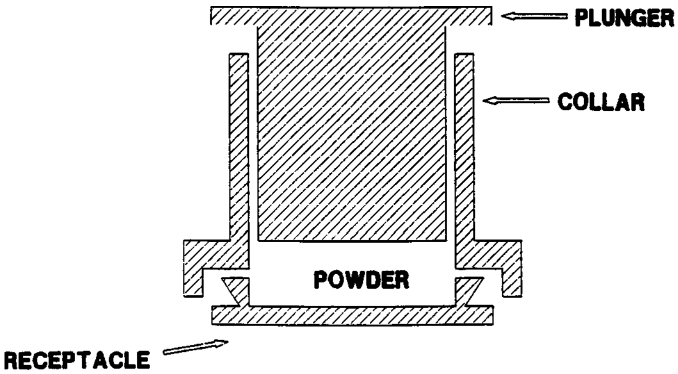
[2] The boldface numbers in parentheses refer to the list of references at the end of this practice.

[3] *Annual Book of ASTM Standards*, Vol 06.01.



NOTE 1—The collar and receptacle should be securely held in place before pressing the powder.

FIG. 1 Example Powder Press

reference standards of diffuse reflectance factor and diffuse radiance factor. The recommended materials are white powders that are pressed into plaques. These plaques provide close approximations to the optical properties of the perfect reflecting diffuser, and may be used to transfer a scale of absolute reflectance to another material or to an instrument.

5. Significance and Use

5.1 All commercial reflectometers measure relative reflectance. The instrument reading is the reflectance factor, the ratio of the light reflected by a reference specimen to that reflected by a test specimen. That ratio is dependent on specific instrument parameters.

5.2 National standardizing laboratories and some research laboratories measure reflectance on instruments calibrated from basic principles, thereby establishing a scale of absolute reflectance as described in CIE Publication No. 44 **(2)**. These measurements are sufficiently difficult that they are usually left to laboratories that specialize in them.

5.3 A standard that has been measured on an absolute scale could be used to transfer that scale to a reflectometer. While such procedures exist, the constraints placed on the mechanical properties restrict the suitability of some optical properties, especially those properties related to the geometric distribution of the reflected light. Thus, reflectance factor standards which are sufficiently rugged and able to be cleaned, depart considerably from the perfect diffuser in the geometric distribution of reflected radiance.

5.4 The geometric distribution of reflected radiance from a pressed powder plaque is sufficiently diffuse to provide a dependable calibration of a directional-hemispherical reflectometer. Although pressed powder standards are subject to contamination and breakage, the directional-hemispherical reflectance factor of pressed powder standards can be sufficiently reproducible from specimen to specimen made from a given lot of powder, so as to allow one to assign absolute reflectance factor values to all the powder in a lot.

5.5 This practice describes how to prepare white reflectance factor standards from a powder in a manner that allows a standardizing laboratory to assign the absolute scale of reflectance to the plaque.

6. Apparatus

6.1 The basic apparatus for producing a pressed powder standard includes a powder press, powder containers and a balance. There are presently two commercial suppliers of powder presses.[4] The press and receptacles can also be made in a local machine shop. A suggested configuration is shown in Fig. 1. The optical surface of the plaque should be pressed against a surface of ground glass or poly(methyl methacrylate) to provide a matte finish on the pressed plaque. Powder receptacles should be at least 5 mm deep for $BaSO_4$ and at least 10 mm deep for PTFE.

7. Reagents and Materials

7.1 *Barium Sulfate*—the barium sulfate should be of the highest purity. It should be specially refined for optical and spectroscopic use **(3)**.

7.2 *Polytetrafluoroethylene*—the PTFE **(4)** should also be specially refined for optical and spectroscopic use but some commercial grades have been found to be acceptable substitutes. **(5)** There is currently no commercial source for small quantities of optical grade PTFE powder. Large quantities (drums) of commercial grade PTFE can be obtained from manufacturers.

8. Procedure

8.1 Store all powdered reflectance standards in tightly capped glass containers. If the powder is purchased in plastic containers, transfer it to a glass container as soon as possible. Before using the powder, place it in a glass blender equipped with stainless-steel or PTFE-coated blades and pulverize to a uniform consistency. Transfer the quantity of powder to be used with stainless steel or PTFE-coated spoons. Perform the whole operation in a draft-free location, away from sources of small particulate contamination, filters, sweaters, windows, ovens, etc. Perform all measurements (weight, height, width, depth, volume, area, etc.) with adequate precision to ensure that the final density is within 5 % of the specified value. The most reproducible standards are made by pressing the powder to a specific density. Thus, determine the mass of the powder to be used from the volume of the receptacle.

8.2 *Barium Sulfate*—Press $BaSO_4$ to a density of 2000 kg/m^3(2.0 gm/cm^3) and a thickness of at least 5 mm. Press several specimens in succession. Select matched pairs to be representative of the contents of the bottle of powder. Keep the pressed plaques in a covered desiccator when not in use. Some suppliers of $BaSO_4$ provide calibration values with each bottle of powder, other suppliers provide only 3 or 4 quality reference checks and a reference to published values of reference standards prepared from the powder. Table 1 gives the 6°/diffuse reflectance factor values for Eastman White Reflectance Standard **(3)**.

8.2.1 Another description of this procedure is found in Ref. **(4)**.

[4] Powder press conforming to ISO 2469 and DIN 5033 is available from Carl Zeiss Canada, Ltd., 45 Valleybrook Drive, Don Mills, Ontario M3B-2S6, Canada, Part Number 505866; and Technidyne Corporation, 100 Quality Avenue, New Albany, IN 47150-2272, Part Number 176601.

TABLE 1 6°/Diffuse Reflectance Factor of Eastman White Reflectance Standard Pressed $BaSO_4$ Powder[A]

Wavelength, nm	Reflectance Factor
300	0.968
350	0.979
400	0.987
450	0.991
500	0.991
550	0.992
600	0.992
650	0.992
700	0.992
750	0.992
800	0.992
850	0.991
900	0.990
950	0.988
1000	0.986

8.3 *Polytetrafluoroethylene*—Press PTFE to a density of 1000 kg/m^3 (1.0 gm/cm^3) and a thickness of at least 10 mm. The spectral reflectance, which is a function of density, has a broad maximum near this density. Prepare several specimens in succession. Matched pairs are selected to be representative of the contents of the container of powder. Keep the pressed plaques in a desiccator when not in use. PTFE has a high dielectric constant and can be very sensitive to airborne particulates. Such contamination can make the material slightly fluorescent and reduce its reflectance in the near-ultraviolet spectral region. Table 2 lists the 6°/diffuse reflectance factor values (**6**) and Table 3 lists the 45°/0° reflectance factor values (**7**) of PTFE as determined by the National Institute of Standards and Technology (NIST).

9. Precision and Bias

9.1 The National Institute of Standards and Technology and the Inter-Society Color Council Project Committee 22, Materials for Instrument Calibration, have carried out collaborative tests to determine the precision and bias of the preparation of PTFE reflectance factor standards **(8)**. The standard deviation of three determinations of the reflectance factor of PTFE by the NIST ranged from 0.0002 to 0.0008 over the spectral range 300 to 1000 nm. The measured reflectances of PTFE from two manufacturers exhibited differences of from −0.002 to +0.004 over the same range with the largest differences near the ends of the range and a constant measurement uncertainty of ± 0.005. From the 9 laboratories participating in the round-robin experiment, 17 specimens were returned. The results are shown in Table 4 for the wavelength range 300 to 1000 nm.

9.2 The National Institute of Standards and Technology carried out collaborative tests to determine the uncertanities in the preparation of PTFE for use as transfer standards of reflectance factor for the 45°/0° geometry. Duplicate pressings

TABLE 2 6°/Diffuse Reflectance Factor of Pressed PTFE Powder[A]

Wavelength, nm	Reflectance Factor
300	0.984
350	0.990
400	0.993
450	0.993
500	0.994
550	0.994
600	0.994
650	0.994
700	0.994
750	0.994
800	0.994
850	0.994
900	0.994
950	0.994
1000	0.994

TABLE 3 45°/0 Diffuse Reflectance Factor of Pressed PTFE Powder[A]

Wavelength, nm	Reflectance Factor[B]
380	1.002
390	1.003
400	1.005
410	1.006
420	1.006
430	1.007
440	1.007
450	1.008
460	1.008
470	1.009
480	1.009
490	1.009
500	1.010
510	1.010
520	1.010
530	1.010
540	1.011
550	1.011
560	1.011
570	1.011
580	1.011
590	1.011
600	1.011
610	1.011
620	1.012
630	1.012
640	1.012
650	1.012
660	1.012
670	1.012
680	1.012
690	1.012
700	1.012
710	1.013
720	1.014
730	1.015
740	1.015
750	1.016
760	1.016
770	1.017

[A] Density = 1000 kg/m^3 and thickness ≥ 5 mm.
[B] Accurate to ± 0.003.

TABLE 4 Average and Standard Deviation of 6°/Diffuse Reflectance Factors of 17 PTFE Plaques Prepared by 9 Laboratories

Wavelength, nm	Reflectance Factor	
	Average[A]	Standard Deviation[B]
300	0.9792	0.0063
350	0.9883	0.0021
400	0.9911	0.0018
500	0.9919	0.0020
600	0.9915	0.0023
700	0.9914	0.0023
800	0.9912	0.0024
1000	0.9910	0.0024

[A] Average Density = 926.2 kg/m ^{3}.
[B] Standard Deviation = 85.7 kg/m^3.

were made of the same PTFE in ten different laboratories. The standard deviations of the measured reflectance factor, as a function of wavelength is shown in Table 5. Table 6 shows the measured reflectance factors, averages, and standard deviations of four different drums of PTFE. The total uncertainty for the transfer is approximately 1 % at wavelengths above 500 nm and approximately 1.5 % at wavelengths below 500 nm in the visible spectral region.

10. Keywords

10.1 bi–directional optical measuring system; hemispherical optical measuring system; integrating sphere; material standards; reflectance and reflectivity; transfer standards

TABLE 5 Difference From the Average of PTFE Powder Pressings Ten Laboratories (A-J) and Two Replicates (1-2)

	Wavelength, nm				
	380	450	600	700	770
A1	0.002	0.004	0.002	0.003	0.004
A2	0.004	0.006	0.003	0.001	0.003
B1	-0.001	0.001	0.000	0.000	-0.007
B2	-0.005	0.000	-0.001	0.000	-0.001
C1	0.009	0.006	0.004	0.004	0.005
C2	-0.005	-0.005	-0.004	-0.006	-0.005
D1	0.001	0.002	0.001	0.001	0.002
D2	0.007	0.004	0.003	0.002	0.002
E1	-0.003	0.002	0.002	0.003	0.004
E2	-0.004	-0.004	-0.005	-0.006	-0.006
F1	0.000	-0.011	-0.004	0.008	0.004
F2	-0.001	0.000	0.001	0.001	0.001
G1	-0.002	-0.004	-0.004	-0.004	-0.005
G2	0.009	0.005	0.002	0.002	0.004
H1	-0.012	-0.006	-0.006	-0.008	-0.007
H2	0.010	0.001	0.000	0.001	0.002
I1	-0.003	0.000	-0.002	-0.001	0.000
I2	0.006	-0.002	0.002	0.002	0.005
J1	-0.010	-0.007	-0.005	-0.007	-0.004
J2	0.003	0.007	0.006	0.008	0.006
Standard Deviation (k=2)	0.012	0.010	0.006	0.008	0.008

TABLE 6 45°/0° Reflectance Factor of Pressed PTFE Powder From Different Drums of PTFE

	Wavelength, nm						
	380	420	470	550	630	700	770
Drum 1	1.005	1.008	1.011	1.013	1.012	1.013	1.016
Drum 2	1.001	1.006	1.009	1.011	1.012	1.012	1.017
Drum 3	0.993	0.999	1.006	1.007	1.010	1.011	1.016
Drum 4	1.007	1.010	1.011	1.012	1.013	1.013	1.017
Mean	1.002	1.006	1.009	1.011	1.012	1.012	1.017
Standard Deviation	0.013	0.010	0.008	0.005	0.002	0.002	0.001

REFERENCES

(**1**) *Publication CIE No. 46—A Review of Publications on Properties and Reflection Values of Material Reflection Standards,* available from USNC/CIE. Request ordering information from Secretary USNC, NIST, Room A317/220, Gaithersburg, MD 20899.

(**2**) *Publication CIE No. 44—Absolute Methods for Reflection Measurements,* available from USNC/CIE. Request ordering information from Secretary USNC, NIST, Room B306/220, Gaithersburg, MD 20899.

(**3**) Grum, F., and Wightman, T. E., "Absolute Reflectance of Eastman White Reflectance Standard", *Applied Optics*, Vol 16, 1977, pp. 2775–2776.

(**4**) *Publication CIE No. 38—Radiometric and Photometric Characteristics of Materials and Their Measurement,* available from US NC/CIE. Request ordering information from Secretary USNC, NIST, Room B306/220, Gaithersburg, MD 20899.

(**5**) Spyak, P. R., Lansard, C., "Reflectance Properties of Pressed Algoflon F6: A Replacement Reflectance-Standard Material for Halon", *Applied Optics*, Vol 36, No. 13, pp. 2963-2970 1997.

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(**7**) Barnes, P. Y., Hasia, J. J., "45°/0° Reflectance Factors of Pressed Polytetrafluoroethylene (PTFE) Powder", *NIST Technical Note 1413*, U.S. Government Printing Office, Washington, D. C., 1995.

(**8**) Weidner, V. R., Hsia, J. J., and Adams, B.," Laboratory Intercomparision Study of Pressed Polytetrafluoroethylene Powder Reflectance Standards," *Applied Optics*, Vol 24, 1985, pp. 2225–2230.